Poster Talks

Neutron Scattering Studies on Materials Science and Engineering in HANARO

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Neutron scattering studies on materials science and engineering performed in HANARO are presented. A 30-MW research reactor had been constructed and reached its first criticality in 1995 at KAERI. Under the government nuclear long-term development program, four instruments -- a high resolution powder diffractometer (HRPD), a four circle diffractometer (FCD), a neutron radiography facility (NRF) and a small angle neutron spectrometer (SANS) were constructed. The residual stress measurement of a ring-and-plug type standard sample and a SUS304 welded plate were performed on an HRPD sample stage using a newly developed high efficiency position sensitive detector. The results showed high spatial resolution and measuring efficiency. The texture of TRIP steel, IF steel and a Zr/Al bilayered sample were measured using FCD, and three dimensional orientation distribution functions(ODF) were obtained from measured complete pole figures. The formability of IF steel was precisely analyzed using obtained ODF and the experimental R-value was in good agreement with the calculated R-value. The preferred orientation correction method by using the recalculated pole figures was applied to the Rietveld profile refinement for highly textured Zr/Al samples, and the precise content of constituents was obtained from refined results. The neutron incoherent scattering for commercial zircaloy tubes was measured using 1-D PSD and HRPD and an infinitesimal amount of hydrogen, about 10 ppm, was quantitatively measured from the incoherent scattering profile. The IF steels were investigated by SANS to determine nano-sized precipitates. The volume fraction and size distribution of nano-sized precipitates was analyzed from SANS pattern.

Chamber for in-situ High Temperature Neutron Diffraction Studies in Controlled Atmospheres

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The experimental conditions required for the study of ceramics can require high temperatures and carefully controlled atmospheres, and there are many examples of ceramics systems for which *in situ* measurements are particularly valuable. In the neutron scattering research field, *in situ* studies of complex systems have become an important area in which to exploit the advantages of neutrons as compared with x-rays, and to justify the construction of more powerful neutron sources. Many of the neutron scattering experiments planned for the upgraded High Flux Isotope Reactor (HFIR) and the Spallation Neutron Source (SNS) will involve time-resolved in-situ studies of materials requiring control of temperature, atmosphere, and other environmental conditions. This presentation will describe the design of a high-temperature environmental chamber (compatible with existing neutron beam lines at HFIR and elsewhere), which is presently under construction. When completed, this chamber will permit crystal structure data, thermal expansion, and phase equilibria to be determined as a function of temperature in a variety of atmospheres.

Materials Science Opportunities Using Focused White-Beam Neutron Techniques

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Local three-dimensional intra-and inter-granular interactions ultimately determine the mechanical behavior of polycrystalline materials. These interactions have been extremely difficult to observe directly and nondestructively. Spatially-resolved 3D probes of the local grain structure, residual stress, and intra- and inter-granular interactions are needed on all length scales ranging from submicron to macroscopic. Newly developed x-ray micro-beam techniques now provide submicron resolution 3-D structural microscopy on the so-called mesoscopic length scale from ~0.1 to 100 μm. The potential for analogous focused neutron Laue diffraction techniques to overlap with x-ray length scales and extend measurements to fully macroscopic length scales is being studied. In this approach, depth-resolved Laue patterns are obtained using high-resolution area detectors and direct depth profiling of diffracted beams. The Laue patterns then provide the local structure, orientation and strain with submillimeter resolution in all three dimensions. The development of this capability would enable 3D materials investigations of plastic deformation, grain growth, stress surrounding fracture cracks, and residual stress characterization of large-grained materials where powder diffraction is not applicable. Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Non-destructive Measurement of residual stress versus depth using Diffraction

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Synchrotron x-ray radiation offers many advantages over conventional sources. One of these is the ability to choose the x-ray energy/wavelength over a wide spectrum. Generally speaking, increasing the x-ray energy increases the depth of penetration. When combined with XRD residual stress techniques, this fact allows the resolution of near surface residual stresses versus depth non-destructively. Traditionally, stress versus depth measurements have been done using either neutron diffraction or a destructive XRD/layer removal technique. The neutron method is excellent for interior measurements, but due to the sampling volume size, it is not appropriate for resolving sharp stress gradients near a surface. The XRD/layer removal technique is capable of resolving these near surface stress gradients, but it is destructive, which is often undesirable.

In this study the x-ray energies selected allowed measurements to be made with the same family of planes, allowing the use of a single x-ray elastic constant generated using conventional methods. The contributions to residual stress at each depth are successively separated from each depth/volume at each energy level via linear numerical inversion method. The residual stress versus depth profiles are then generated for two 0.4% carbon steel samples with different surface treatments: 1) forged and shot peened, and 2) induction hardened and ground. The results will be compared to the XRD/layer removal and neutron measurements.

Analysis of Residual Stresses in Bent Composite Tubes and Welded Air Port Panels Using XRD and Finite Element Methods

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Cracking in composite tubes (SA210 clad with 304L) used to fabricate primary air ports in kraft recovery boilers is well-known but not well understood problem for boiler operators. Unlike cracking in composite floor tubes, where the cracks terminate at the cladding/substrate interface, cracks in composite air port tubes sometimes penetrate into the carbon steel layer which can lead to catastrophic in-service failure of the boiler. From the research performed on composite floor tubes it is now commonly accepted that differences in the thermal expansion characteristics of the 304L and the SA210 combined with the elevated in-service operating temperature of the boilers can yield residual stresses sufficient to promote stress corrosion cracking (SCC) in the 304L. Typically, these cracks terminate at the 304L/SA210 interface. In-service inspection of clad tubes in air port panels, however, has revealed that cracks in the clad layer sometimes extend into the carbon steel. Further consideration of air port tubes and floor tubes reveals that both manufacturing processes and operating conditions experienced by each type of tubing are typically different. For example, the air port tubes are bent during manufacturing and can be exposed to localized frequent in-service thermal transients 100 degrees C above the nominal operating temperature. In this study some of the aforementioned manufacturing and operating variables will be investigated using X-ray and neutron diffraction in conjunction with finite element (FE) modeling . In particular, residual stresses and strains from two specimens will be presented: (1) a single bent composite tube (prior to welding) designed for a primary air port opening, and (2) an air port panel removed from service with cracking present. Finite element simulated welding stresses in an air port panel will be combined with the bent tube stress measurements in order to characterize the "as fabricated" stress state of an air port panel. Subsequently, these stress values will be used as initial conditions in another FE model that simulates the in-service temperature environment. Finally, neutron strain measurements from the air port panel will be used to validate the FE results. Research sponsored by the US Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S. Dept. of Energy under contract DE-AC05-00OR22725. T. Ely was also supported in part by an appointment to the Oak Ridge National Laboratory Postdoctoral Research Associates Program administrated jointly by the Oak Ridge Institute for Science and Education for the Oak Ridge National Laboratory.

Prediction of Distortion and Residual Stresses
in Heat Treated Components

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The heat treating process essentially establishes the final quality of the components during manufacturing processes. Often, a significant percentage of the produced components have to be scrapped when they do not meet specification. The wasted time, energy, and material impair the manufacturer's efficiency and competitiveness. Using a computational tool to predict the distortion and residual stress in heat-treated components has a tremendous potential benefit. This project aims at developing an accurate model with reliable data for predicting the distortion and residual stress in heat treated components. The models can be used to parametrically study the effects of different quenching strategies to minimize distortion or residual stress. The three major technical tasks include material characterization, thermal boundary conditions determination, and system validation. The project is focusing on the study of two material systems, 4142 and IN718, each with different geometries that are representative of industrial components, and two different quenching media appropriate for each material. System validation is being done by comparing the results of distortion from numerical simulations with measurements from the processed components. Measurement of residual stresses in the heat treated components by neutron diffraction is in progress. After the successful performance of the project, accurate prediction of the distortion and residual stress in heat treated components will be possible.

Grain-Orientation-Dependent Residual Stress Measuring by Pulsed Neutron Source

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Normally, when diffraction is used to measure the *macro* residual stress in materials, a linear 'd-space' v.s. $\sin^2\psi$ relationship is expected. However, for materials that have undergone large deformation, a so-called non-linear phenomenon, i.e. an oscillating 'd-space' v.s. $\sin^2\psi$ distribution is often observed even in samples where the stress in the radiated layers is known to be bi-axial. The physical nature of this non-linear behavior has been mainly attributed to two factors: the influence of crystallographic texture on the diffraction elastic constants and the existence of Type II stress which is grain orientation dependent. The inter-granular or Type II stress in polycrystalline materials in caused by stress or strain incompatibility between grains having different crystallographic orientations during mechanical or thermo-mechanical deformation. Thus, a study of the grain orientation dependence of the residual stress not only enables an unambiguous assessment of the macro-residual stress but also helps to answer fundamental questions such as how the grain-to-grain interactions occur in a polycrystalline material during and after deformation. Furthermore, investigations of the grain-orientation-dependent residual stress provide valuable information concerning the history of thermo-mechanical treatments in engineering materials. Recently, a method was developed for experimental evaluation of the grain-orientation-dependent residual stress from neutron or x-ray diffraction measurements. Analogous to texture representation by the orientation distribution function (ODF), the concept of the stress orientation distribution function (SODF) was defined as a continuous stress field in the Eulerian space. The SODF can be directly constructed from the lattice strain distributions along various specimen directions, i.e., the strain pole figures (SPFs). Pulsed neutron sources using time-of-flight technique provide an effective tool to map the SPFs, which can simultaneously monitor the shift and broadening of a large number of hkl-planes. In this poster, we describe the methods of measuring the generalized pole figures using a pulsed neutron source and constructing the grain-orientation-dependent stress from those diffraction data. The validity of this method is demonstrated through measurements of the grain-orientation-dependent strain in a deformed Interstial-free steel sheet using GPPD at the Intense Pulsed Neutron Source in Argonne National Laboratory.

Towards 0.1 mm spatial resolution

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One of the design goals for VULCAN, the SNS engineering diffractometer, is the ability to measure spatial changes with 0.1 mm resolution (in one-dimension). The major challenge here is to define 0.1 mm wide incident and diffracted beams. Because the targeted applications often involve the use of large samples or special environment, slits cannot be used for this purpose. In this paper, methods to achieve 0.1 mm spatial resolution are outlined. For the incident beam, a new compact focusing device is proposed. The device is made of a stack of bent silicon wafers, each having a reflective multilayer (supermirror) deposited on one side and a neutron absorbing layer on the other side. The optimal design to minimize the optical spatial aberrations is discussed and Monte-Carlo simulation results are presented. The present design for the diffracted beam focuses on imaging. Preliminary testing results with Bragg Mirror imaging will be presented.

Microstructure evolution in irradiated pyrochlore:
***in-situ* TEM and HRTEM observation**

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Pyrochlore compounds, $A_2B_2O_7$, have remarkable physical properties, including piezoelectricity, ferroelectric and ferrimangetism, giant magneto-resistance and metallic/electric/ionic conductivity, due to the extensive chemistry compositions and various structural disordering. These various physical properties coupled with the controlled microstructure make pyrochlore a very attractive candidate in numerous technological applications: catalysis, oxygen sensor, magnet, cathode and electrolyte materials for solid oxide fuel cell and the potential actinide host phase in nuclear waste. Ion beam technique has been used to tailor the properties of pyrochlore compounds by engineering the microstructure and structural disorder extent for the specific applications. In this study, TV-rate *in-situ* TEM and *ex-situ* HRTEM observations were conducted in irradiated pyrochlore compositions: $Gd_2Ti_2O_7$ and $Gd_2Zr_2O_7$. The radiation effects, microstructure evolution of pyrochlore structure subjected to heavy ion irradiation and the mechanism to control the response of pyrochlore compositions under heavy radiative environment was understood.

Deep Inelastic Neutron Scattering:New Terrain

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As the momentum transfer increases, the neutron scattering function S(q,w) approaches the impulse approximation limit, in which it is determined entirely by the momentum distribution of the scattering particles. Recent work at ISIS has shown that it is possible to measure the momentum distribution for hydrogen in solids and liquids with unprecedented accuracy, and in some cases to extract a Born-Oppenheimer potential and spatial wavefunction for the hydrogen. We will describe the method of doing that and give some recent examples of its application.

"SMARTS - A spectrometer for residual strain and in situ loading measurements"

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In-Situ Neutron Diffraction Investigation of Phase Transformations in Nickel Base Superalloys

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Microstructure evolutions in nickel base superalloy welds are far from equilibrium due rapid cooling conditions. Due to rapid cooling rates, phase stability and partitioning characteristics between γ (FCC) and γ' ($L1_2$ ordered precipitates) are complex. The focus of this research was to characterize the high-temperature stability of a nonequilibrium microstructure in a CM247CC nickel base superalloy. One of the important microstructural parameters is the lattice misfit between γ and γ' phases. To measure these changes rapidly in bulk samples, neutron scattering experiments were performed using the GEM diffractometer at ISIS, UK. Large quantities of diffraction data (over 1500 spectra in each experiment) were collected *in-situ*. The lattice mismatch was found to change from $\sim 4\times10^{-4}$ to $\sim 8\times10^{-4}$ while the sample was held isothermally at 1000°C. In the same period, the (110) peak intensity of γ' did not change significantly. These results indicate that there was no growth or dissolution of γ' phase at this temperature and only changes in the lattice mismatch occurred due to diffusion of alloying elements between γ and γ' precipitates. The implication of these results towards the development of welding and heat treatment methodologies will be highlighted.

Characterization of C_xH_y intermediates formed during methane activation on supported metal catalysts by in-situ neutron vibrational spectroscopy (NVS)

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Activation of methane and conversion to higher hydrocarbons are extremely challenging and technologically important process, and will greatly benefit from an enhanced understanding of surface species formed on the catalyst surface during the methane activation process. NVS, which can be employed for investigating supported catalysts at industrially relevant pressure/temperature conditions, is highly sensitive towards hydrogenous species, is not limited by selection rules and allows accurate quantitative estimations; thus represents an ideal technique for characterizing surface hydrocarbonaceous species on supported catalysts. This NVS investigation provides the first experimental evidence for the formation of methylidyne (CH) , vinylidene (CCH_2) and ethylidyne (CCH_3) species from methane on supported metal catalysts. The figure (shown below) shows the vibrational modes for the various species (CH, CCH_2 and CCH_3) formed on the Ru/Al_2O_3 surface after methane decomposition at 523 K. The studies also reveal that the vinylidene species were more stable at higher reaction temperatures. Interestingly similar species (to that observed on supported Ru catalysts) were also observed on supported Ni catalysts. Combining these results with our earlier studies on Ru model catalysts (high resolution electron energy spectroscopy) a mechanism has been proposed for the non-oxidative methane homologation on alumina supported Ru catalysts. These studies clearly show the effectiveness of NVS to probe reaction mechanisms/pathways and thereby to provide an enhanced understanding of the catalytic process.

The Engineering Program at the NIST Reactor

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SANS Measurement of Hydrides in Uranium

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SANS scattering is shown to be an effective method for detecting the presence of hydrogen precipitates in uranium. High purity polycrystalline samples of depleted uranium were given several hydriding treatments which included extended exposures to hydrogen gas a at two different pressures at 630°C as well as a furnace anneal at 850C followed by slow cooling in the near absence hydrogen gas. All samples exhibited neutron scattering that was in proportion to the expected levels of hydrogen content. While the scattering signal was strong, the shape of the scattering curve indicated that the scattering objects were large sized objects. Only by use of a very high angular resolution SANS technique was it possible to make estimates of the major diameter of the scattering objects. This analysis permits an estimate the volume fraction and mean size of the hydride precipitates in uranium.

Trends in Sample Environment Technology

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Researchers rely on a variety of sample environment (SE) devices to measure and control parameters such as temperature, pressure, and magnetic field. There is increasing need for advanced SE systems that control multiple parameters, incorporate automation, provide wider operating ranges, and allow a variety of *in-situ* experiments. Many advanced SE systems have emerged, and many more are on the horizon, thanks to the efforts of SE teams at various neutron scattering facilities, university-based teams, and vendors worldwide. Here we present a sampling of these efforts from institutions worldwide.

Neutron Powder Diffraction Studies of Structure II Hydrate Formed from a Methane + Ethane Gas Mixture

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Neutron powder diffraction data collected at the high resolution powder diffraction beamlines BT-1 (NIST) and HRPD (JAERI) confirmed that hydrate samples with a gas composition of 82 mol% CH_4 and 18 mol % C_2H_6 crystallize as structure II hydrates. In all data sets ice was present as a secondary phase. The structure has been modeled using rigid-body constraints to describe CH_4 molecules located in the sixteen small polyhedral cavities and a 50/50 mixture of CH_4/C_2H_6 located in the eight large polyhedral cavities of a deuterated host lattice. The data collected at BT-1 allowed for full refinement of lattice parameters, atomic coordinates, and atomic displacement parameters. The larger magnitude of atomic displacement parameters of the guest molecules in the larger cages indicate more positional disorder than shown by the CH_4 molecules in the small cages.